

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of:

Toshihisa YOKOYAMA, Ken-ichi NODA, Katsuhira IMAI

and Minom IMAEDA

Ser. No.: 09/854,924

Group Art Unit: 1765

Filed: May 14, 2001

Examiner:

Song, M.

Confirmation No.: 7936

PROCESS AND APPARATUS FOR PRODUCING AN OXIDE SINGLE

CRYSTAL

RULE 132 DECLARATION OF KATSUHIRO IMAI

- I, Katsuhiro IMAI, hereby declare and state that:
- I received a Doctor of Science degree in Mineralogy in March, 1993 from the University of Tokyo.
- 2. I have been employed by NGK Insulators, Ltd., the assignee of the above identified application, since April, 1993. During my employment with NGK, I have been involved with research and development of: single crystal materials at the Research Laboratory of NGK Insulators, Ltd., from April, 1993.
- I have reviewed the prosecution history of the above-identified application, 3. particularly the Final Office Action mailed January 15, 2003 and the Advisory Action mailed March 28, 2003. I have also reviewed Imaeda et al. and Ciszck et al., the applied prior art of record.

- 4. In the Advisory Action, the Examiner is taking the position that Applicants have not provided evidentiary support for the arguments that: 1) silicon crystals typically have a higher coefficient of thermal conductivity compared to that of oxide single crystals; and 2) silicon crystals typically have a lower coefficient of thermal expansion compared to that of oxide single crystals.
- Kisohen II (Handbook of Chemistry, Fundamental Chemistry Volume II), 4th edition,
 Maruzen, Tokyo (in Japanese), a data sheet obtained from the INRAD, Inc., website
 http://www.inrad.com/pdf/Inrad_datasheet_LNB.pdf (a printout of which is attached hereto).
 oxide single crystals typically have a coefficient of thermal conductivity of 4W/m·K and
 coefficients of thermal expansion of 14.1x10⁻⁶/K(//a) and 4.1x10⁻⁶/K(//c), whereas silicon
 crystals typically have a coefficient of thermal conductivity of 148W/mK and a coefficient of
 thermal expansion of 4.15X10⁻⁶/K. Therefore, those of ordinary skill in the art understand
 that silicon crystals have a higher coefficient of thermal conductivity and a lower coefficient
 of thermal expansion in comparison to oxide single crystals.
- 6. The Imacda et al. reference relates to oxide single crystals and that reference correctly recognizes that it is undesirable to have too high of a cooling rate after growing the oxide single crystals. Although Imacda et al. do not specifically address cooling oxide single crystals at the liquid-solid crystal interface, based on the disclosure in Imacda et al., I would also conclude that one should not use a cooling gas to directly cool the oxide single crystal liquid-solid crystal interface. This is because the temperature at the liquid-solid crystal interface is necessarily higher than the temperature of the grown crystal body, and thus, blowing a cooling gas directly on the oxide single crystal solid-liquid crystal interface

would necessarily produce a greater rate of temperature change at the hotter liquid-solid crystal interface than would be realized if the same cooling medium were blown on the cooler crystal body. Therefore, for the same reasons that it is undesirable to have too high of a temperature gradient in the grown crystal body after crystal growth, as specifically disclosed by Imaeda et al., it is also undesirable to have too high of a temperature gradient at the liquid-solid crystal interface.

- step of directing a cooling medium onto the liquid-solid crystal interface portion. Ciszek's method is successful because of the relatively higher coefficient of thermal conductivity and the relatively lower coefficient of thermal expansion compared to those of oxide single crystals (see paragraph 5 above). On the other hand, blowing a cooling medium onto the liquid-solid crystal interface of Imaeda's oxide single crystal would not necessarily produce the benefits disclosed in Ciszek, because of the relatively lower coefficient of thermal conductivity and the relatively higher coefficient of thermal expansion of oxide single crystals. Therefore, skilled artisans would not conclude that oxide single crystals could withstand Ciszek's liquid-solid crystal interface cooling treatment, which, again, is designed for silicon crystals, because oxide single crystals have a lower coefficient of thermal conductivity and a higher coefficient of thermal expansion in comparison to silicon crystals.
- 8. Based on my academic credentials and work experience, I consider myself to be one of ordinary skill in the art. The claimed invention was not obvious to me at the time it was made. Nor do I believe that the claimed invention would have been obvious to others of ordinary skill in the art absent our discovery.

9. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date 9, 2003

Katsuhiro Imai

Lithium Niobate (LINbO₂)

PHYSICAL PROPERTIES

Chemical Formula

LiNbO₃ congruently melting³

Crystal Symmetry and Class

trigonal, R3c

Point Group

3ті

Lattice Constants². a = 5.15052(6) Å c = 13.86496(3) Å

Density²

4.648(5) g/cm³

Moh's Hardness

Fracture Toughness³

じ-1800

0.67 MPam^{1/2}

x-face : y-face

1.07 MPam^{1/2} 1.17 MPam^{1/2}

Elastic Compliance 4 at Constant Polarization (S_P) and at Constant Field (S_E) and Temperature Dependence 5

(⊤Pa) ⁻⁷	(TPa)-1	(10 ⁻⁴ /°K)
Se13= 4.76	S _{E11} = 5.78	$(1/S_{E11})dS_{E11}/dT=1.66$
Spag= -0.50	S _{E12} = - 1.01	$(1/S_{E12})dS_{E12}/d1=0.28$
Sp13= -1.20	S ₅₁₃ = -1.47	$(1/S_{E13})dS_{E13}/dT=1.94$
S _{P14} = 1.02	S _{E14} = -1.02	$(1/S_{E14})dS_{E14}/dT=1.33$
S _{P33} = 4.19	S ₅₃₃ = 5.02	$(1/S_{\pm 22})dS_{\pm 22}/dT = 1.60$
S _{P44} - 9.3	S _{E44} - 17.0	(1/S _{E44})dS _{E44} /dT-2.05
Spec= 10.5	Srec= 13.6	$(1/S_{ccc})dS_{ccc}/dT=1.43$

Stiffness⁴ at Constant Polarization (C_P) and at Constant Field (C_E) and Temperature Dependence⁵

(CPa)	(CPa)	(10 ⁻¹ /°K)
$C_{P11} = 219$	$C_{E11} = 203$	(1/C _{E11})dC _{E11} /dT=-1.74
C _{P12} = 37	C ₅₁₂ = 53	$(1/C_{012})dC_{013}/d1 = -2.52$
$C_{P13} = 76$	C _{E13} = 75	(1/C _{E13})dC _{E13} /dT=-1.59
Cp14= -15	C _{F14} = 9	(1/C _{F14})dC _{F14} /dT=-2.14
C _{P22} - 252	C _{E22} - 245	(1/C _{E22})dC _{E22} /dT1.53
C _{P44} - 95	C _{E44} - 60	$(1/C_{E49})dC_{E99}/dT - 2.04$
C _{P66} - 91	C _{E88} - 75	$(1/C_{E00})dC_{E00}/dT=-1.43$

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OPTICAL AND ELECTRO-OPTICAL PROPERTIES
                  Optical Symmetry
                                                      uniaxial negative
            Optical Transmission
                                                          0.400 μm - 5.0 μm
                          Sellmeier Equation Constants<sup>13</sup>
                   \Pi = (A + B/(\lambda^2 + C) + D\lambda^2)^{1/2}; \lambda in microns
  n<sub>o</sub> A=4.9048 B=0.11768
                                                C= -0.0475
                                                                         D= -0.027169
                            B-0.099169
                                                    C--0.044432 D- -0.02195
     n<sub>e</sub> A~4.582
                      Calculated Refractive Index Values<sup>12</sup>
            n_o(1.064 \mu m) = 2.2322; n_c(1.064 \mu m) = 2.1560
            n_n(2.050 \mu m) = 2.1949; n_n(2.060 \mu m) = 2.1243
            n_{\rm e}(3.500~\mu{\rm m}) = 2.1405; n_{\rm e}(3.500~\mu{\rm m}) = 2.0788
             Photoelastic Strain Coefficients at Constant Field<sup>11</sup>
                           p_{71} = -0.026
                                                    P31 ≈ 0.17
                                                    \rho_{22} = 0.07
                          \rho_{12} = 0.08
                          \rho_{13} = 0.13
                                                   \rho_{41} = -0.151
                          p_{14} = -0.08
                                                 p_{44} = 0.146
Temperature Variation of Refractive Index ^{13} for \lambda = 1.0~\mu m = 4.0~\mu m dn/dT = 3.3 x 10^{-5} /°C
                               dn./dT - 37 x 10 5/°C
                       Nonlinear d Coefficients 12,20
                                   d_{22} = 2.4 \text{ pm/V}
                                   d<sub>31</sub> = -4.52 pm/V
d<sub>33</sub> = 31.5 pm/V
                     Effective Nonlinear Optical Coefficient
                         d_{eff} = d_{33} \sin \theta - d_{22} \cos \theta \sin 3\Phi
                   Electro Optic Coefficients @ 0.633 µm<sup>23</sup>
            r_{13}^{T} = 10 \text{ pm/V}

r_{22}^{T} = 6.8 \text{ pm/V}

r_{33}^{T} = 32.2 \text{ pm/V}
                                          r_{13}^{S} - 8.6 \text{ pm/V}

r_{22}^{S} = 3.4 \text{ pm/V}

r_{33}^{S} = 30.8 \text{ pm/V}

r_{51}^{S} = 28 \text{ pm/V}
            r<sub>32</sub>' = 32.2 μ...
r<sub>51</sub>' = 32 pm/V
      Variation of Electro Optic Coefficient rzz with Wavelength<sup>22</sup>
And Calculated Half-wave Voltage For 9mmx9mmx25mm Q-Switch
                               V1/4 - 2 d/ (4 n3 1 r22)
            1.064 \, \mu m = 5.6 \, pm/V
                                                           1.55 kVolts
              1.318 \mu m = 5.4 pm/V
                                                             2.02 kVolts
              1.55 \ \mu m = 5.3 \ pm/V
                                                             2.44 kVolts
            2.10 \ \mu m = 5.2 \ pm/V
                                                           3.45 kVolts
              2.79 \mu m = 5.1 \rho m/V
                                                             4.78 kVolts
              2.94 \muin = 5.1 pm/V
                                                             5.DB kVolts
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3 J/cm² @ 10 nsec

Damage Threshold²

THERMAL AND ELECTRICAL PROPERTIES

Melting Point7

1240° C

Curie Temperature⁶

1145° C

Thermal Conductivity9

. 1. W/m°K

Thermal diffusivity⁶

9 x 10⁻⁷ m²/sec

Specific Heat®

0.633 J/q^K

Thermal Expansion 10

 $\alpha_{\rm g} = 14.1 \times 10^{-6} / {\rm °K}$ $\alpha_{\rm c} = 4.1 \times 10^{-6} / {\rm °K}$

Resistivity¹⁴

2 x 1010 Ω - cm @ 200° C

Dicloctric Constants¹⁸

Loss tangent 19 @400 °C

1 and =0.0006

Tanδ =0.001

Typical Polish Specifications

Wavefront Distortion: λ/8 @ 633 nm

Flatness:

λ/10 @ 633 nm

Parallelism:

1 arcseconds

Scretch - Dig:

10 - 5

Description

Lithium niobate is a ferroelectric material suitable for a variety of applications. Its versatility is made possible by the excellent electro-optic, nonlinear, and piezoelectric properties of the intrinsic material. It is one of the most thoroughly characterized electro-optic materials, and crystal growing techniques consistently produce large crystals of high perfection.

Applications that utilize the large electro-optic coefficients of lithium niobate are optical modulation and Q-switching of infrared wavelengths. Because the crystal is nonhygroscopic and has a low half-wave voltage, it is often the material of choice for Q-switches in military applications. The crystal can be operated in a Q-switch configuration with zero residual birefringence and with an electric field that is transverse to the direction of light propagation. Because piezoelectric ringing can be severe, piezoelectrically damped designs can be very useful. The damage threshold of the intrinsic material at 1.06 microns with a 10 nsec pulse is approximately 3 J/cm². With appropriate AR coatings, a surface damage threshold of 300-500 MW/cm² can be achieved for the same conditions.

Applications that use the large nonlinear d coefficient of LiNbO3 include optical parametric oscillaton, difference frequency mixing to generate tunable infrared wavelengths, and second harmonic generation. With a broad spectral transmission, which ranges from 0.4 μm to 5.0 μm with an OH absorption at 2.87 μm , a large negative birefringence, and a large nonlinear coefficient, phasematching is an effective way to generate tunable wavelengths over a broad wavelength range.

Lithium niobate is particularly effective for second harmonic generation of low power laser diodes in the 1.3 to 1.55 µm range.

For infrared generation by difference frequency mixing, the peak power limit is considerably lower than for 1.064 μ m, being about 40 MW/cm². Efficiencies for difference frequency mixing generally are smaller than sing efficiencies with KDP or BBO, which is due to the lower peak powers that can be tolerated by the crystal and the fact that the longer wavelength photons that are generated in the process are less energetic. Typical powers for 10 nanosecond long pulses with 5 mm diameter beams are 30 mJ/pulse of 0.640 μ m minus 40 mJ/pulse of 1.064 μ m to produce 2.5 mJ/pulse at 1.54 μ m, and 32 mJ/pulse of 0.640 μ m to produce 0.25 mJ/pulse at 3.42 μ m.

INRAD offers lithium niobate in a variety of configurations. Standard cuts are available as OPO crystals, Q-switches, difference frequency mixing crystals, autocorrelation crystals, and optical waveguide wafers.

Please consult an INRAD sales engineer for assistance in crystal selection and packaging.

At INRAD, all crystal growth, orientation, fabrication, pollshing, and testing of LiNbO $_3$ is done at one site so that you are assured of complete traceability and satisfaction with every crystal that you purchase.

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5 - 2 膨張率と圧縮率

七ルシウス型度がりにおよび8における長さお上び休機。 と、それぞれにいいおよび」、ひとすれば、一般に越野盗事の

5-2-1 団件単体の独局混率むよび体閥張率

および体験薬率」はそれぞれ

TSZ SALBON . .. 生た温度 むおよび むにおける値をそれぞれ し かままび h. m.とすれば、B.と B.との門の平均県最高平 B. お上び平 心体産品中 ローはそれぞれ

$$\beta_{m} = \frac{1}{J_{v}} \frac{I_{a} - I_{1}}{\theta_{o} - \theta_{1}} \tag{0-3}$$

$$a = \frac{1}{v_0} \frac{v_0 - v_1}{g_2 - g_1} \quad ... \quad (5 - 6)$$

で与えられる」。さらに特密な目的に対しては

$$v = v_0 \left(1 + \alpha (\theta + \alpha 2\theta^2 + \alpha_3 \theta^3)\right)$$

$$v=v_{\bullet}\left(1+\tilde{a}(\theta+ad\theta+\tilde{a}_{\bullet}D^{\bullet})\right)$$
 (5-6)
で走わされる。 なお写方性の動質なは、 $\mu=3\beta$ である。

 $(5\cdot 1)$

3 - 29 MAX MAT COM SANTE ON

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^{1) &}quot;Landolt-Bimaciti Zablenwerte und Frankfirmen una Physik, Chemie, Astronomie, Geophysik und Technik". 6 Auft., II Band, I Tell. S. 378~64& Springer-Variety (1971).

1 体导频率 a triat 病 (全方).

175

・2-2 化合物の組制版率および体施版率で

表 5-20 网络化疗器的性型变体 2.5.1.1/1 电应变体点 (1)

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[&]quot;Lemdolt-Borestein Zahlenismte und Kunktionen aus Flyzik. Chemies Astronomies Geophysik und Technik", a Auft. II Band. 1 Teil. 5. 409-778, Springer Verlag (1971).,

^{≠ 2} 配收

在 6 - 65 图体学体内的配子中***** E/Wing K**

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y ,.	. 13.5	10.5	9.50	107	100	125	15,2	Se (2 c)		נפ	10.5	0.08	4,22	1		1
r 	2.25	177	14.8	. 149	139.,	1¢£	1 T.R	Si	200		884	268	2.78	5.36 BE.D	50.0	1
<u>;</u>	372	130	24	80.3	-694	1E,7	32.5	See	,	7.52	7.35	113.	E		1	27.2
100	63.6	£7.A	12.4	. 40.E	(F7.D)	1	f	30	111		ES	73.2	1	133	DH.20	1:00
1	19,0	11.5	8.98	928	10.1]	Ta	. 7	- 1	59.2	37.5	57.5	57.8	53	(40.5)
:	81E	232	96.8	- 232	433	- 227.1	37.4	Тъ	1 1	8.7.	ננ	2.98	ins	1 -1.2	🐃	60.2
, e4	25.1	25.0 ·	SIA	23.0	· 424	£7.0	20.7	Telf c)	•	1	259	silb.	2.15	- 254	220	I
2	35.4	320	2A 0	(A.94)	(9.84)	(127)	aln	772	53	1.2	£8.B	40.6	451	103	59.4	1
,	7.77	7.62	120	130	134	١.,	·	Ţ.	40	ul.	37.6	94.5	21.5	20.0	19.4	51.5 20.7
	1	- 1]	· DAM	(0.114)	(0.058)		T	. ₽	. 5	55.6	48,4	461	43.8		} ===
- 1	104	97.E	89.7	#1_7	74.E	42.4	52.4	Tm '	10.	.	13.5	15.2	163			l
1	472	172	153	147	ME	135	126	ע	18.	9	71.7	25,1	27.5	29.5	35.6	43.9
- 1	112	107	204	202	(52.0)	(40.4)	(30.30	v	40.	,	35.7	31.5	31.5	32.1	95,9	23.9 . 283.6
. 1	0.42	9.78	iya	13-6	ZLO	.124	. وعد	У	16,		12.7	15.5	16.2	16.5	20.3	23.4 23.4
1	235	114	EEI	76.5	72.1	L50,9)	(60.00)	አየ .			· 1	al.i	365	34.1		÷
, 1	20.9	18.9.	17.2	10%		-		Zn	213	11	24	PCI	ובו	116	100.00	(EZ.2)
;-	375 LDS	165			1:28	147	(an	=	43.7	7 :	552°	اعم	22.7	21.6	20.9	, 23, 7
	CLAS	5 70	7.17	7.95			ĺ			1		- 1			_	,; •

1 元中の()を付けた数値は最累認相の位である。

2 東安名に何けた 1c. グェチとけ、単位品配名の c軸に単立・c 軸に平行などを示す。ただし、C(高品)については、ATJ 気気の加熱は形体の を変名に何けた上に、Jaかどは、単独品配名ので軸に生む、GMLでいるというである方に対する記憶(上)、平行(J)を示し、L(Lyn)は動分所是論を示す。

日本	22			R R	T/K				造度T/K							
	100	200	300	400	300	· 700	1000	E \$	100	200	300	400	500	700	1000	
	0.64	11	1.8	3 1.51	1.60	1,92	2.87	MgU SiO ₄	270	.94	<u>so</u>	48.1.	32			
へ YTEX 77個)・' P C 7 ム	0.50	0.91	nri	121	1.16	T 62		(石英上に前)	29 20 <i>0</i>	16A 8.5	10.6	7.6	6.D	45 3.i		
(CO 6)	5_52	4.74	3.99	3.03	3.45	3.19	297	ThO. TiO.		٠	122	18,2	81	5.5	3,7	
' 'ファイク) 対 あ)	450	52	40	32.4	24.2	324	10.5	(ルチルタの数)	23.5 16.9	13.7	104 7:4	8.5 6.0				
	133	55 524	36 373	25.4 196	. 20,2. 146	32.6 87 ·	2.9 17	(多穀品)。 DOz	6.0	E.G	8 <i>A</i> 82	7.0	5.8 6.5	77 11	a.e.	

Coming # 19 p=2.226 3 g cm - Coming # 19 p=2.201 g cm -